A comparative study of selected properties of ProRoot mineral trioxide aggregate and two Portland cements

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Abstract

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Aim To compare solubility, microhardness and radiopacity of ProRoot mineral trioxide aggregate (MTA) with two Portland cements (PC: CEM I and CEM II).

Methodology Solubility: for standardized samples ($n = 12$/group) ring moulds were filled with the cements. These samples were immersed in double-distilled water for 1 min, 10 min, 1 h, 24 h, 72 h, and 28 days. Mean loss of weight was determined. Microhardness: five samples of each cement were produced. All samples were loaded with a diamond indenter point with a weight of 100 g for 30 s. Radiopacity: five samples per cement were produced. These samples were tested according to the ISO standards to compare their radiodensity to that of an aluminium step wedge (1–9 mm). Differences between the three materials with respect to their solubility, microhardness and radiopacity were analysed using ANOVA and Student–Newman–Keuls.

Results After 28 days MTA was of low solubility (0.78%) compared with CEM I (31.38%) and CEM II (33.33%). At exposure times >1 min the two PCs were significantly more soluble than MTA ($P < 0.05$). The microhardness for MTA was significantly higher (39.99 HV; $P < 0.001$) compared with the two PC (CEM I: 16.32 HV; CEM II: 13.51 HV). MTA was significantly more radiopaque (5.34 mm Al) than CEM I (3.32 mm Al) and CEM II (2.52 mm Al) ($P < 0.05$), whereas CEM I was significantly more radiopaque than CEM II ($P < 0.05$).

Conclusions Mineral trioxide aggregate displayed superior material properties than both Portland cements.

Keywords: densitometry, radiopacity, solubility, Vickers microhardness.

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Introduction

ProRoot mineral trioxide aggregate (MTA) is an 'endodontic repair cement' and was applied for patent and introduced in the mid 1990 by Torabinejad & White (1995, 1998). According to the manufacturer's information (Dentsply Tulsa Dental, Tulsa, OK, USA) the indications for ProRoot MTA are: root-end filling, apexification, repair of perforations, and direct pulp capping.

Some authors described ProRoot MTA as a composition of ordinary Portland cement with added bismuth oxide for radiopacity (Estrela *et al.* 2000, Funteas *et al.* 2003, Camilleri *et al.* 2005). Hence, several studies compared biological effects of ProRoot MTA with Portland cements. MTA and Portland cement were not cytotoxic when evaluated *ex vivo* (Ribeiro *et al.* 2005), both released arsenic well below the level considered to be harmful (Duarte *et al.* 2005), and both

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showed no difference in cell reactions (Saidon *et al.* 2003) and similar antimicrobial activity (Estrela *et al.* 2000, Sipert *et al.* 2005). Dentine tubes filled with MTA, Portland cement and calcium hydroxide implanted in rat subcutaneous connective tissue caused very similar tissue reactions (Holland *et al.* 2001b). Implanted in mandible bone of guinea pigs both materials were well tolerated (Saidon *et al.* 2003). Wucherpfennig & Green (1999) capped the pulps in rats either with ProRoot MTA or Portland cement. The effects on pulp cells were very similar and comparable to calcium hydroxide. These results were confirmed in direct pulp capping studies in the teeth of dogs (Holland *et al.* 2001a, Menezes *et al.* 2004).

As the biological effects of both materials were identical it was suggested that in dental practice ProRoot MTA might be replaced by cheaper Portland cement (available in do-it-yourself stores) (Wucherpfennig & Green 1999, Saidon *et al.* 2003, Menezes *et al.* 2004, Duarte *et al.* 2005, Ribeiro *et al.* 2005).

Beside the described similarities marked differences between Portland cement and ProRoot MTA were described comparing some chemical and physical surface and bulk material characteristics (Dammaschke *et al.* 2005): in ProRoot MTA the amount of gypsum is approximately half of the Portland cements. The composition of ProRoot MTA consists of less toxic heavy metals (Mn and Sr), less chromophores (Fe^{3+}), and less aluminium and potassium species. In contrast to Portland cements, ProRoot MTA contains about 17–18 wt% (=2 atom%) bismuth. Portland cements are composed of particles with a wide range of size whereas ProRoot MTA showed an equal and smaller particle size (Dammaschke *et al.* 2005).

Surprisingly, beside the above mentioned biological comparison between ProRoot MTA and Portland cement comparative studies analysing the chemical and physical properties of the two materials are rare in the dental literature. There is no study directly comparing ProRoot MTA and Portland cements concerning selected properties of the materials such as solubility, radiopacity and microhardness. These are important factors as an endodontic material should be more radiopaque than its surrounding structures, gutta-percha or other filling materials. Also it must be insoluble to avoid leakage and should be as hard as possible to avoid dislodgement from the dentine wall (Torabinejad *et al.* 1995).

Therefore, the aim of the present study was to compare solubility, microhardness and radiopacity of ProRoot MTA with the Portland cements type CEM I and CEM II.

Materials and methods

Solubility test

White ProRoot MTA and two commercially available Portland cements were included in this study. According to the European Standard EN 197-1 (2000), one of the Portland cements was classified as type CEM I (Teutonia Portlandzement, EN 197-1-CEM I 32,5 R, Teutonia Zementwerk, Hannover, Germany) and the other as type CEM II (Felsenfest Portlandkalksandsteinzement, CEM II/A-LL 32,5 R EN 197-1, Spenner Zement, Erwitte, Germany) Portland cement. White ProRoot MTA was obtained from Dentsply DeTrey (Konstanz, Germany, Batch: 02093081).

Solubility was determined in double-distilled water. The solubility tests recorded weight loss of the test specimens, and followed to a great extent the methodology of the International Standard ISO 6876 (2001). Stainless steel ring moulds having an internal diameter of 20.0 mm (±0.1 mm) and a height of 1.6 mm (±0.1 mm) were used for sample preparation. All moulds were cleaned with acetone in an ultrasound bath for 15 min. Thereafter a copper wire was fixed at each mould in order to hang the specimens in a glass dish in such way that the surfaces did not touch and the materials remained undisturbed in the dish. All moulds were weighed three times prior to use (accuracy ±0.0001 g).

ProRoot MTA was mixed according to the manufacturer's instruction. The Portland cements CEM I and CEM II were mixed in the same powder to liquid ration as ProRoot MTA (1 g cement powder: 0.3 mL distilled water). The moulds were placed on a glass plate and filled to slight excess with the mixed material avoiding air entrapment. All samples were left to set on a grating in an incubator at 37 °C for 24 h and 95% relative humidity. Excess material was then trimmed to level the surface of the mould using silicone carbide paper (600 grit). From each material, 72 samples were prepared, which were divided into six groups of 12, for immersion in water for 1 min, 10 min, 1 h, 24 h, 72 h, and 28 days. Thus, a total of 216 samples were prepared for this study.

Prior to the immersion of the samples, all materials in their moulds were weighed (Sartorius type 1801 MPS, Göttingen, Germany) three times and the average reading was recorded. All weight measurements were in grams and recorded to four decimal places.

Six samples of each material in its mould were immersed in a fresh 160 mL aliquot of liquid at 37 °C

(±1 °C) a time for one day and subsequently in fresh 160 mL aliquots at weekly intervals. The specimens were placed in an airtight dish (7 × 10.5 × 8 cm) with 95–100% relative humidity such that both surfaces of each sample were freely accessible to the liquid. There was no agitation of the dish. As controls, 24 empty sample moulds together with the copper wire were immersed in water for 28 days, and any changes in weight were recorded.

Samples of cements were removed from the dish after the specified immersion period using a pair of tweezers, touching only the metal mould. Samples were washed with 3 mL of double-distilled water and allowed to dry for 24 h at 37 °C in an oven. The specimens were placed on a grating in such a way that only the metal moulds touched the grating. Thereafter the samples were weighed three times and the mass of the cements was determined to the nearest 0.0001 g. The amount of material removed from the specimen was recorded as the difference between the original weight of material and its final weight to the nearest 0.0001 g. This difference in mass was calculated as a percentage of the original weight of the material, recorded to the nearest 0.001%.

Differences between the three materials with respect to their solubility were analysed using a one-way analysis of variance (ANOVA) and the *post hoc* Student–Newman–Keuls test for all pairwise comparisons ($P < 0.05$).

Vickers microhardness

For measurements of Vickers microhardness (HV), ProRoot MTA was mixed according to the manufacturer's instruction. The Portland cements CEM I and CEM II were mixed in the same powder to liquid ration than ProRoot MTA (1 g cement powder: 0.3 mL distilled water). All mixed cements were brought into silicon moulds with a size of 10 mm in length, 5 mm in width and 5 mm in height. To avoid the inclusion of air the cements were vibrated for 1 min with a vibration intensity of 6000 min^{-1} (KV 36, Wassermann Dental-Maschinen GmbH, Hamburg, Germany). Subsequently, the samples were covered with parafilm ('M'-Laboratory Film, American CAN Company, Greenwich, CT, USA) and left to set in an incubator at 37 °C for 24 h and 95% relative humidity. Five samples of each cement were produced, 15 in total. One side was then trimmed using silicone carbide paper (600 grit).

For the measurement of the microhardness one polished cement surface of each sample was loaded with a diamond indenter point (Durimet, Wetzlar, Germany) with a weight of 100 g for 30 s to produce a stamp mark in a homogeneous region of the cement surface. The diamond indenter produced one impression with two orthogonal diagonals equal in length, which were measured immediately after discharge. The microhardness was calculated as follows:

$$HV = 0.102 * \frac{F}{A} \approx 0.1891 * \frac{F}{d^2}$$

$$A = \frac{d^2}{2 * sin\frac{136}{2}}$$

where F = load in Newton's, 0.1891 = Vickers constant; d = arithmetic mean of the two diagonals, A = impression surface in mm^2, HV = Vickers hardness.

Each cement sample was measured at five defined points resulting in 25 measurements per cement and a total of 75.

The data were treated statistically by analysis of variance (ANOVA) and *post hoc* Student–Newman–Keuls test at a level of significance of $P < 0.05$.

Radiopacity

According to International Standard ISO 6876 (2001) stainless steel ring moulds having an internal diameter of 10.0 mm (±0.1 mm) and a height of 1.0 mm (±0.1 mm) were used for sample preparation. Five samples per cement were produced and allowed to set for 24 h. From each cement one sample was placed on a dental X-ray film (Kodak Insight Dental Film, Film Speed E, LOT 104 3665, Kodak, Rochester, NY, USA) together with an aluminium step wedge (1–9 mm). The X-ray exposures were made using a Sirona Heliodent DS X-ray unit (Bensheim, Germany) with a Sirona tube and a 2.5 mm aluminium filter (Bensheim, Germany) added. The tube voltage was 60 kV and the current 7 mA. The exposure time was 120 ms with a constant source-to-film distance of 21 cm. The films were developed, fixed, and dried in an automatic processor (Dürr-Dental XR 24 Nova, Dürr, Bietigheim-Bissingen, Germany).

The densities were measured with a densitometer (Darklight duo ref; Medset, Hamburg, Germany) with a measuring range $D = 0$ up to $D > 4.5$ and accuracy for $D < 3$ +0.01.

Differences between the three materials with respect to their radiodensity were analysed using analysis of variance (ANOVA) and the *post hoc* Student–Newman–Keuls test for all pairwise comparisons ($P < 0.05$).

Results

Solubility

There was no change in the weight of empty moulds after immersion in water after 28 days. The results for all materials are shown in Table 1.

ProRoot MTA was of low solubility. The weight loss after 28 days' immersion in water was 0.78%. Thus, ProRoot MTA was virtually insoluble. The weight loss of the two different Portland cements after 28 days' immersion ranged from 31.38% to 33.33% (Table 1). At exposure times >1 min the two Portland cements were significantly more soluble than ProRoot MTA ($P < 0.05$). There were no significant differences between the weight loss of the two Portland cements at all exposure times ($P > 0.05$).

Vickers microhardness

The mean microhardness for ProRoot MTA was with 39.99 HV approximately 2.5-fold higher than for CEM I (16.32 HV) and CEM II (13.51 HV), respectively (Table 2). The differences between ProRoot MTA and CEM I and CEM II were highly significant ($P < 0.001$), whereas no significant difference was obtained between the two Portland cements ($P = 0.619$).

Radiopacity

The analysis showed a statistical difference among the three groups (Table 3). CEM I was significantly more radiopaque than CEM II ($P < 0.05$). ProRoot MTA was significantly more radiopaque than both Portland cements ($P < 0.05$).

Table 2 Surface microhardness of the three materials

Cement	Mean	SD	95%-confidence interval for the mean	
CEM I	16.32	4.17	14.60	18.05
CEM II	13.51	3.69	11.98	15.03
ProRoot MTA	39.99	16.65	33.12	46.87

Given are the mean, SD and the 95%-confidence intervals of the Vickers hardness.

Table 3 Radiopacity of the three materials

	Millimetres of aluminium	
	Mean	SD
CEM I	3.32	0.33
CEM II	2.52	0.27
ProRoot MTA	5.34	0.13

Given are the mean and SD as millimetres of equivalent thickness of aluminium.

Discussion

Solubility

In the International Standard ISO 6876 (2001) the procedure to determine the solubility of set root canal sealer in water is described. The solubility tests performed in the present study followed to a great extent the methodology of this International Standard because ProRoot MTA can be used as root-end filling getting direct contact with periapical tissue like sealers.

However, while weight loss of the test specimens was recorded by determining the decline in mass of the material samples after storage in water, as already described by some authors (McComb & Smith 1976, Ørstavik 1983, Kazemi *et al.* 1993, Ono & Matsumoto 1998), the International Standard suggests that the increase in weight of the dish in which the samples have been placed (residue method) should be ascertained as the amount of material removed from the specimens (Higginbotham 1967, Kaplan *et al.* 1997, International Organization for Standardization 2001). The specimens were weighed in order to avoid an underestimation of the material going into solution. In order to enhance the accuracy of the measurements, one sample was used for just one immersion period, thus undesirable weight loss of the cements because of repeated drying and immersion was excluded.

Table 1 Solubility of the three materials

	1 min		10 min		1 h		24 h		72 h		28 days	
Material	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
CEM I	0.04	0.13	0.78	0.76	25.91	0.26	26.41	2.73	28.71	4.43	33.33	5.23
CEM II	0.03	0.06	0.76	0.77	21.37	5.83	25.84	4.56	29.50	7.45	31.38	4.01
ProRoot MTA	0.13	0.14	0.13	0.14	0.54	0.26	0.69	0.27	0.71	0.26	0.78	0.29
P-value	0.164		<0.05		<0.001		<0.01		<0.001		<0.01	

Given are the mean percentages with SD of weight loss for each material and for each immersion period.

It has to be kept in mind that with regard to the strict definition of the physicochemical term solubility, the test used in the present study measured the elution of water-soluble material, but not the solubility (Wilson 1976). Solubility of a solid is the situation where a pure chemical compound is in thermodynamic equilibrium with its solution (Wilson 1976). Moreover, it has to be taken into account, that measuring weight differences of the cement specimens may also record disintegration processes that may not be the result of dissolution (Wilson 1976, Ørstavik 1983). For instance, particles of the material may fall out from the cement structure during storage in the liquid (Wilson 1976, Ørstavik 1983). Furthermore, water uptake may compensate for dissolved material (Ørstavik 1983, Caicedo & von Fraunhofer 1988, Kazemi *et al.* 1993).

It was found that both Portland cements were significantly more soluble than ProRoot MTA. Under the conditions of this *in vitro* study ProRoot MTA can be described as nearly insoluble. This finding is in accordance with a report by Torabinejad *et al.* (1995). The marked differences between ProRoot MTA and CEM I and CEM II may be due to differences in chemical surface composition after setting reaction of the cements. ProRoot MTA consists of less sulphur and potassium species but has increased calcium content at the materials surface (Dammaschke *et al.* 2005). These differences in surface composition may explain the reduced solubility of ProRoot MTA: the higher sulphur content in Portland cement is connected with the higher gypsum content compared with ProRoot MTA. It can be speculated that the higher gypsum content in Portland cement is one reason for the increased solubility. Moreover, the bismuth oxide added in ProRoot MTA is virtually insoluble in water (Fridland & Rosado 2003).

Vickers microhardness

Regarding microhardness it was found that ProRoot MTA was significantly harder than both Portland cements. The measurement of the microhardness was undertaken with 5-mm thick samples to simulate clinical application. Matt *et al.* (2004) recommended 5-mm thick ProRoot MTA as an apical barrier, which was significantly harder than a barrier of 2 mm (Matt *et al.* 2004). In addition the minimal thickness for ProRoot MTA given in the literature as root-end filling material is 3 mm (Lamb *et al.* 2003) and for apexification 4 mm (Giuliani *et al.* 2002).

The hardness of ProRoot MTA depends on the size of the cement particles, the water-to-powder ratio, the temperature and humidity, and the amount of air entrapped in the mixture (Torabinejad *et al.* 1993). Because all cements where mixed and treated in the same way the only variable that might has influenced the microhardness is the particle size of the cement powder. The differences in the particle size of the three materials tested are of great importance for the mechanical characteristics of the bound cements. With a similar particle size a higher mechanical strength is designed by a reduced spreading in grit size (Locher *et al.* 1973), which could be observed in ProRoot MTA. That the particle size of ProRoot MTA is more equal and smaller than the Portland cements tested here was proved by Dammaschke *et al.* (2005). In addition the reduced microhardness of the two Portland cements could be understood because of reduced potassium content in ProRoot MTA (Dammaschke *et al.* 2005), as potassium is known to decrease the mechanical properties of cements (Strunge *et al.* 1985a,b).

According to Ryge *et al.* (1961) the microhardness of intact dentine is about 70 HV and thus approximately twofold higher than that of ProRoot MTA. Moreover, it has been reported that in an acidic environment (pH 5) as in periapical inflammation the microhardness of ProRoot MTA samples were even significantly lower than in neutral environment (Lee *et al.* 2004). Summarising these observations and taking into account that the microhardness of both Portland cements was substantially lower than that of ProRoot MTA, CEM I and CEM II seam to be unsuitable for long-term clinical use.

Radiopacity

Root-end filling materials must be radiopaque in order to be able to evaluate the quality of the filling. It is known that the radiopacity of a 1-mm thick mineralized tissue is equivalent to that of 1 mm of aluminium (Manson-Hing 1961). Therefore, according to the ISO standard 6876 (International Organization for Standardization 2001), a radiopacity of 3 mm of aluminium is requested for root filling materials.

According to the present results, ProRoot MTA was significantly more radiopaque than both Portland cements (Table 3). That is not astonishing because ProRoot MTA contains about 2 atom% bismuth (Dammaschke *et al.* 2005). Bismuth oxide was added to improve the radiodensity. Only ProRoot MTA and CEM I complied with the requirement of the ISO standard (International Organization for Standardization 2001).

In general, the present findings concerning the radiopacity are in agreement with previous studies in as far as for grey MTA an radiodensity of 6.4 mm of aluminium (Laghios *et al.* 2000) respectively 7.17 mm of aluminium (Torabinejad *et al.* 1995) has been reported. Comparing these data and those of the present study with the radiopacity of other dental materials, it becomes obvious that MTA is less radiopaque than Super-EBA (9.9 mm Al), IRM (9.3 mm Al), gutta-percha (11.0 mm Al) or amalgam (15.6 mm Al) (Laghios *et al.* 2000) but in the same range as zinc oxide–eugenol based root canal sealers. The later ones have been reported to show a radiopacity between 5.1 mm and 9.1 mm of aluminium (Camps *et al.* 2004).

Conclusions

The Portland cements CEM I and CEM II were significantly more soluble, reached less microhardness values and were less radiopaque than ProRoot MTA. These differences in the analysed product properties could be explained by the differences in chemical composition. With regard to these properties, it is questionable if ProRoot MTA can simply be substituted by the cheaper Portland cement for endodontic treatment as recommended in several publications (Wucherpfennig & Green 1999, Saidon *et al.* 2003, Menezes *et al.* 2004, Duarte *et al.* 2005, Ribeiro *et al.* 2005).

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